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350 MARAGING STEEL PROCESSED BY POWDER METALLURGY

SAUL ISSEROW
MATERIALS APPLICATION DIVISION

September 1976

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ARMY MATERIALS AND MECHANICS RESEARCH CENTER
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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER AMMRC TR 76-29	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) 350 MARAGING STEEL PROCESSED BY POWDER METALLURGY		5. TYPE OF REPORT & PERIOD COVERED Final Report
		6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) Saul Isserow		8. CONTRACT OR GRANT NUMBER(s)
9. PERFORMING ORGANIZATION NAME AND ADDRESS Army Materials and Mechanics Research Center Watertown, Massachusetts 02172 DRXMR-K		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS D/A Project: 1T162105AH84 AMCMS Code: 612105.11.H8400 Agency Accession: DA OE4813
11. CONTROLLING OFFICE NAME AND ADDRESS U. S. Army Materiel Development and Readiness Command, Alexandria, Virginia 22333		12. REPORT DATE September 1976
		13. NUMBER OF PAGES 20
14. MONITORING AGENCY NAME & ADDRESS(if different from Controlling Office)		15. SECURITY CLASS. (of this report) Unclassified
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Approved for public release; distribution unlimited.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Maraging steels	Ductility	Heat treatment
High strength steels	Metal powder	Hydrogen embrittlement
Toughness	Extrusion	Mechanical properties

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

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ABSTRACT

Rods and tubes of 350 maraging steel were extruded from rotating electrode process powder and from cast-and-wrought bar stock from two sources. Samples from the rods and tubes received various heat treatments and were then subjected to tests of tensile behavior and impact resistance. The results of these tests and metallographic examination are presented and discussed as they are affected by factors such as material source and heat treatment. Consideration is given to scale-up to larger components and to means of achieving higher strength maraging steel.

CONTENTS

INTRODUCTION

Processing of prealloyed powders offers several advantages in highly alloyed materials such as the maraging steels. Alloy powders provide the usual benefit of improved homogeneity relative to cast material. In addition, preparation of powder by rapid cooling of molten globules imparts finer distribution of microstructural features in the powder. The resulting reduction in scale of distribution of compositional and microstructural features can have a marked effect on mechanical behavior of metals as manifested, for instance, in tensile and compressive strengths, ductility, impact resistance, and appearance of fracture surfaces. Thus, prealloyed powder has led to significantly altered behavior - generally, but not automatically improved - in alloy systems such as titanium,[1] magnesium,[2] aluminum,[3] and nickel-base superalloys.[4] Application of this approach to eliminating segregation in maraging steel is the subject of a pending patent application by AMMRC personnel.[5]

Different methods are available for large-scale preparation of prealloyed powders. For rapid quenching, two methods have been applied to a variety of alloys. One method, atomization, depends on one of the inert gases to disperse a stream of molten metal into globules which freeze as they dissipate heat to the gas. The other method, the rotating electrode process (REP) invented by A. R. Kaufmann,[6] depends on formation of molten metal at the tip of a rapidly rotating electrode when an arc is maintained between this and amother electrode, the latter generally being a nonconsumable tungsten rod. The molten globule is whirled off by centrifugal force and freezes in flight. The choice of powder preparation method for a specific situation is determined by factors such as powder size and morphology required, reactivity of the molten alloy with crucible or nozzle materials, ease of electrode preparation, and economic considerations.

This report compares the results obtained from rods and tubes extruded from REP powder of 350 maraging steel with similar extrusions from cast-and-wrought (c/w) stock.

BACKGROUND

The maraging steel family offers a remarkable combination of high strength and toughness, even at strengths beyond 300 ksi. In premium service requirements such as shells or aircraft, considerable benefit accrues from even a modest increment in either strength or toughness. Previous work has indicated that the powder approach offers a means of enhancing one of these properties without compromising the other. In previous programs on 300 and 400 maraging steels,[7,8]

1. ABKOWITZ, S. *High Strength Wrought Weldable Titanium Alloy Mill Product Manufacture.* U.S. Patent 3,343,998, September 26, 1976.
2. ISSEROW, S., and RIZZITANO, F. J. *Microquenched Magnesium ZK60A Alloy.* International Journal of Powder Metallurgy and Powder Technology, v. 10, no. 3, July 1974, p. 217.
3. JACOBSON, L. A., PIERCE, C. M., and COOK, M. M. *Microstructures of Powder and Conventionally Processed 7075 Aluminum Alloy.* Air Force Materials Laboratory, AFML-TR-71-240, December 1971.
4. FRIEDMAN, G., and KOSINSKI, E. *High-Performance Material from a Hot-Worked Superalloy Powder.* Metals Engineering Quarterly, v. 11, no. 1, February 1971, p. 48.
5. RIZZITANO, F. J., and ABRAHAMSON, E. P., II. *An 18% Ni-Co-Mo Maraging Steel Having Improved Toughness and its Method of Manufacture.* Patent Pending.
6. KAUFMANN, A. R. *Method and Apparatus for Making Powder.* U.S. Patent 3,099,041, July 30, 1963.
7. ABRAHAMSON, E. P., II. *Processing and Properties of 18Ni Maraging Steel by Powder Metallurgy.* Army Materials and Mechanics Research Center, AMMRC TR 73-4, February 1973. AD 758 439.
8. ABRAHAMSON, E. P., II. *Processing and Properties of 13Ni (400) Maraging Steel by Powder Metallurgy.* Army Materials and Mechanics Research Center, AMMRC TR 74-14, June 1974.

Abrahamson investigated rods extruded from REP powder and demonstrated that the best combination of strength and toughness is achieved by direct aging of the extruded rod without intermediate solutionizing or homogenization. He compared his results with data available for commercial cast-and-wrought materials and claimed the superiority of the powder-derived material. Reassessment of his data relative to that for commercial stock, however, shows that his material displayed properties within the ranges reported for commercial material. Evaluation of the benefits of powder calls for concurrent characterization of identically processed cast-and-wrought stock, preferably from the same source as the REP powder. This course has been followed in the current program on 350 maraging steel.

Maraging steel powders can also be prepared by gas atomization. Snape and Veltry[9] extruded such 350 powder at higher temperatures than used by Abrahamson. The extruded rods were homogenized and aged before testing. The strengths were somewhat lower (Y.S. 320 ksi; U.T.S. 332 ksi) than for c/w material, but very high impact strengths (23.5 to 27 ft-lb) were observed. These values were attributed to benign heterogeneity in the extruded rod, as shown also by the woody fracture.

In a broad study, Van Swam, Pelloux, and Grant investigated different methods of powder preparation and consolidation for 300 maraging steel.[10] Powder-derived material was superior to conventional material in tensile properties but fracture toughness and fatigue were not improved. Commercial stock was improved in both tensile properties and toughness by grain refinement through heavy hot rolling.

Work with elemental or partially prealloyed powders has yielded inferior mechanical properties compared with fully prealloyed powders (see, for example, Ref. 8), and is therefore not cited here.

APPROACH AND SCOPE

This program has concentrated on 350 maraging steel (nominal composition 17.5 Ni, 12.5 Co, 3.8 Mo, 1.7 Ti, 0.15 Al; in the actual powders Ni and Mo are increased, Ti and Al are decreased, see Table 1). Rods and tubes were extruded

Table 1. CHEMICAL ANALYSIS OF 350 MARAGING STEEL (WEIGHT PERCENT)

Element	4-1/2 in. RCS Bar Source	4-1/2 in. RCS Bar AMMRC	3-in.-dia Bar (Electrode Stock) Source	3-in.-dia Bar (Electrode Stock) AMMRC	REP Powder AMMRC
Ni	18.65	18.44	18.39	18.54	18.43
Co	12.03	12.44	12.03	12.18	12.28
Mo	4.70	4.57	4.78	4.68	4.64
Ti	1.38	1.47	1.35	1.68	1.56
Al	0.10	0.14	0.13	0.08	0.16
C	0.008	0.008	0.011		0.016

9. SNAPE, E., and VELTRY, F. J. *The Properties of 18Ni 350 Maraging Steel Produced from Elemental and Prealloyed Powders.* International Journal of Powder Metallurgy, v. 8, no. 4, October 1972, p. 193.
10. VAN SWAM, L. F., PELLOUX, R. M., and GRANT, N. J. *Properties of Maraging Steel 300 Produced by Powder Metallurgy.* Powder Metallurgy, v. 17, no. 33, 1974, p. 33.

under conditions guided by Abrahamson's experience with 300 and 400 maraging steels.[7,8] Both configurations were prepared from powder and also from c/w stock from two sources. Two types of powder were used in each case, REP and gas-atomized. The gas-atomized powders gave unsatisfactory extrusions, which were not processed further. Both tube and rod stock were subjected to several aging treatments before mechanical testing, and were tensile tested. The rod stock was also used for Charpy impact tests.

PREPARATION OF MATERIALS

Powder

Standard 10-inch-long electrodes for conversion to powder by REP were machined from commercial 3-inch-diameter bar. Each rod was provided with a 2-1/2-inch-diameter hub, 2 inches long. The remaining length was machined to a diameter near 2.7 inches to maximize the powder yield while remaining within the 16-pound limit specified for each electrode. Twenty-one electrodes gave 261 pounds of powder. The following sieve analysis was obtained for the powder.

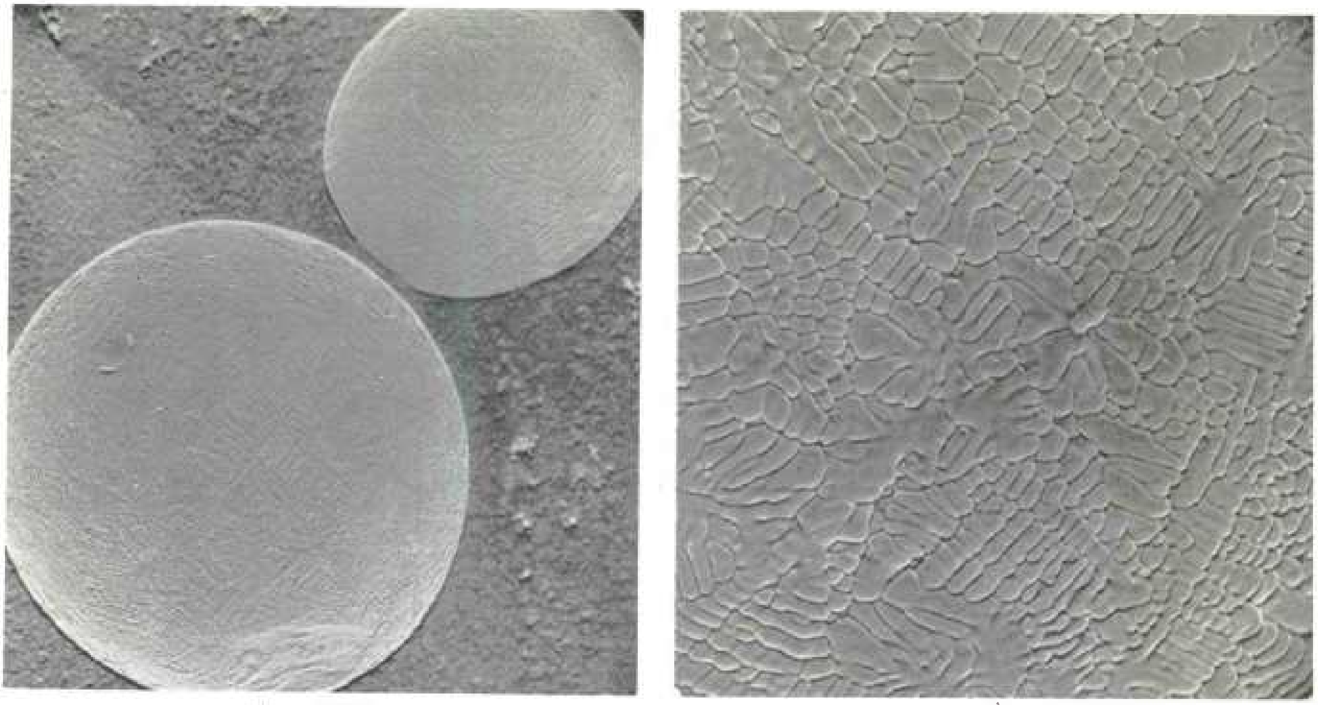
Sieve, U.S. series:	35	45	60	80	120	170	230	325	PAN
Microns:	500	354	250	177	125	88	63	44	<44
Percent retained on screen:	0	1.71	11.79	28.44	22.54	21.17	7.99	5.27	1.09

Powder was used directly for the extrusion billets except that the fines (-325 mesh) were first screened out. Chemical analyses of the powder and the c/w stock at AMMRC gave the results listed in Table 1, along with the analyses reported by the vendors. The surface of the powder is seen in the scanning electron microscope (SEM) photomicrographs at different magnifications in Figures 1a and 1b. The fine aligned dendrites are seen in a section through the powder in Figure 2. Microhardness measurements with a 100-gram load averaged 398 HK corresponding to HRC 39 or less, indicative of the solutionized structure.

One tube and one rod were extruded from each of two different lots of gas-atomized powder. Unfortunately, both lots proved to be contaminated, resulting in defective extrusions containing excessive numbers of inclusions and some cracks. For this reason the processing of these powders is mentioned only incidentally in the remainder of the report.

Extrusions

A set of six tubes and a set of five rods were extruded at different times on a 1400-ton press under essentially the same conditions, see Table 2. All billets (except for Rod 5) consisted of mild steel cans in which the maraging steel stock was contained. The cans were evacuated, outgassed at 800 F, and sealed. The powder charges were weighed and showed a packing density of about 68% of theoretical for REP powder, versus about 63% for gas-atomized. The powder billets were not subjected to any separate prior compacting step. The upsetting of the preheated



Mag. 225X Mag. 1125X

Figure 1. SEM photomicrographs of 350 maraging steel powder by rotating electrode process (REP).

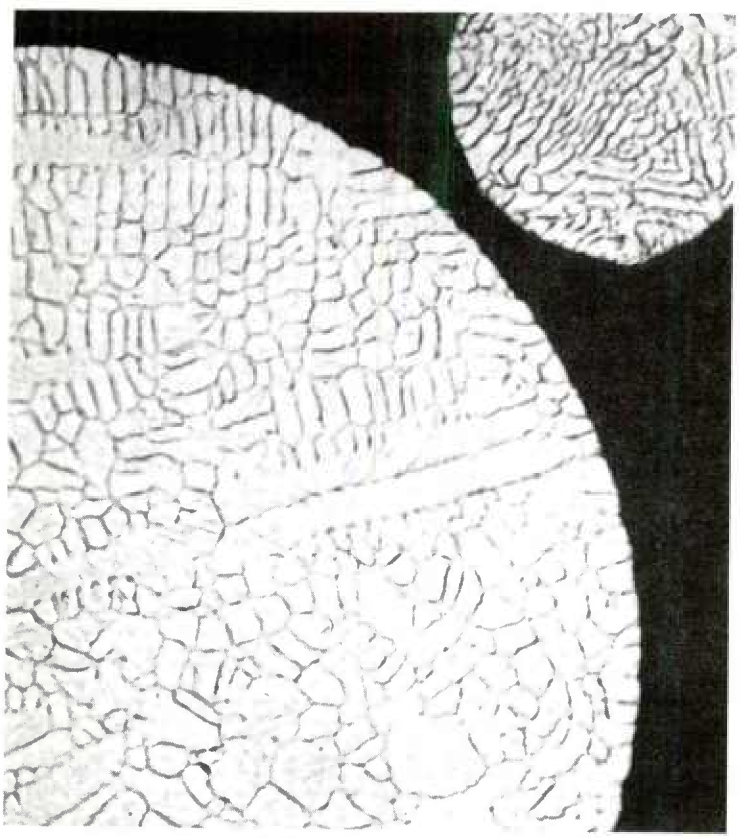


Figure 2. Optical micrograph of section of 350 maraging steel powder by rotating electrode process (REP). Mag. 1000X

Table 2. EXTRUSION OF 350 MARAGING STEEL BILLETS

Material Iden.	Material	Temp deg F	Upset Force Tons	Upset K_u* tsi	Running Force tons	Running K_r* tsi
Tubes - Tool diameter:		liner, 4.655"; mandrel, 1.4"; die, 2.0"; R = 9.6				
A	Cast/wrought 4-1/2-in. RCS bar	1660	1220	34.2	1070	30.5
B	Front: c/w 3-in. electrode stock, upset to 4 in. Rear: same as A	1660	1150	32.8	1040	29.6
C	Gas-atomized powder	1660	1210	34.5	1050	29.9
D	Gas-atomized powder	1660	1250	35.6	1075	30.6
E	REP powder	1660	1275	36.3	1150	32.8
F	REP powder	1660	1300	37.0	1175	33.5
Rods - Tool diameter		liner, 3.625"; die, 1.0"; R = 13.1				
1	REP powder	1400	1125 - stalled (117 tsi stress on 3-1/2-in. ram versus usual 110 tsi limit)			
		1550	1110	41.8	1005	37.9
2	3-in. c/w electrode stock	1650	1010	38.1	950	35.8
3	REP powder	1650	980	36.9	950	35.8
4	Gas-atomized powder	1650	960	36.2	910	34.3
5	4-1/2-in. c/w RCS bar†	1650	1050	39.6	950	35.8

*P=F/A=K ln R
†Extruded bar, omitting mild steel canning and evacuation

billet within the extrusion tools was considered to provide the prior compaction. All billets except for Rod 1 were preheated to 1650 F and the extrusions were rapidly water quenched.

Tubes with nominal dimensions of 1.83 in. OD x 0.20 in. wall were sought in the series of six extrusions. Two billets were prepared from each of the three types of 350 maraging steel stock: cast and wrought; gas-atomized powder; and REP powder. The second tube (B) had different c/w stock in the front and rear halves (see Table 2). The extrusion forces, especially at the upset or breakthrough of the billets, approached the capacity of the press. For example, the final billet, containing REP powder, needed 1300 tons for the upset. These high forces compounded problems arising from the small diameter of the mandrel and the difficulty of maintaining

lubrication between the billet and the mandrel. Each of the four powder-containing billets thus manifested a deficiency associated with the mandrel. Each type of powder gave one billet in which the load was sufficient to cause failure of the mandrel under tension, so that the inward deformation of the billets was not controlled by the mandrel; the resulting tubes (C and F) were unsatisfactory. Each type of powder did give one satisfactory tube (D and E) which, however, was bound on its mandrel so that the tube did not clear the die but had to be cut in the press before it could be placed in quench tank. The two billets of solid stock (c/w) extruded with no difficulty and were quickly water quenched.

Rods with diameters near one inch were obtained in the series of five extrusions. Four rods represented different starting materials extruded under identical conditions, two powders and two c/w bars. (One of these bars was extruded bare since it was large enough for extrusion in the same press liner without any can.) An additional REP powder billet was prepared for extrusion at lower temperature. After this billet stalled in an attempt to extrude at 1400 F, it was turned down and extruded about a week later at 1550 F. The other four extrusions, all at 1650 F, were uneventful. The high reduction ratio (13.1) and the high extrusion constants (K_u and K_r) necessitated ram stresses very close to the limit of 110 tons per square inch.

Post-Extrusion Processing

Surface cleanup before heat treatment was necessary to remove lubricants, canning, or oxidation product. For rods, machining is the simplest means, especially for test specimens. For tubes, chemical means were sought to overcome any difficulty in machining the inside and to conserve the rather thin wall (about 0.2 inches.) To reduce the generation of fumes and also the possiblity of hydrogen pickup by the core metal, a substantial portion of the outer can was machined off before removal of the balance by acid. Initially 10-inch lengths of tubing were pickled in a 1:1 aqueous solution of concentrated nitric acid. For a subsequent section, E3, the 1:1 solution used concentrated hydrochloric acid, which left a thicker wall, indicating that the nitric acid had attacked the maraging steel at a slower rate than the mild steel jacket.

The tubes posed another problem in preparation of test specimens: matching of the curvature to a grip or fixture. It was decided to flatten tube sectors for flat serrated grips. This procedure was first checked on a strip cut from tube B prepared from c/w stock. Strips about 3/4 inch wide were cut from cleaned 9-inch lengths from the tube. At this stage the tubing was still relatively soft (about 32 to 35 HRC). The strips were flattened at room temperature in a forging press with subsequent straightening on an anvil. Substantial machining close to final dimensions is best performed at this stage, before the aging treatment for rods as well as for tubes. Similarly, the Charpy specimens were milled from the rods, aged, and then notched.

Final aging warranted investigation because of differences between recommendations from various sources and also possible differences in the response of powder-derived materials. The following heat treatments, designated A through D, were used.

A. Age 6 hours at 950 F
B. Age 3 hours at 950 F
C. Age 6 hours at 900 F
D. Solutionize one hour at 1500 F; age 6 hours at 950 F

Heat treatment D added solutionizing before final aging mainly because this STA treatment (solution treat and age) is the standard sequence for conventional material. All heat treatments were in air (no protective atmosphere in the furnace) and the samples were removed from the furnace and left in air for cooling.

Since the results for the tubes showed that treatments B and C produced under-aged material, these treatments were included only for Rod 1, on the chance that the lower extrusion temperature might modify its aging response.

MECHANICAL TESTS

The rods were used to prepare standard round tension specimens (0.505-in. gage diameter) and standard Charpy V-notch specimens. The flattened tube sectors were used to obtain standard flat tension specimens, but the width of the gage section was reduced from 0.5 to 0.3 in. to reduce the load required in the test.

The results of the tests are summarized in Table 3 for the tubes and Table 4 for the rods. Elongation data are not included for the tubes since almost all the specimens gave very low values as a result of failure outside the gage marks. This behavior is conceivably related to the flattening of the tube sectors. Yet an effect due to straining in the flattening operation seems to be ruled out by the similarity of properties for strips aged directly (heat treatment A) and strips resolutionized at 1500 F before aging (heat treatment D).

METALLOGRAPHY

The powder specimens were metallographically prepared using the following etchant: 1 g $CuCl_2$, 150 ml water, 50 ml concentrated HCl. The fine aligned dendrites are seen in Figure 2.

Sections from the tubes and rods were examined, both as-extruded and heat-treated. All sections were cut longitudinally, that is, parallel to the extrusion direction, to permit observation of fibering or banding. The etchant was 5 g $FeCl_3$, 2 ml concentrated HCl, about 300 ml ethyl alcohol.

The as-extruded tubes are seen in Figure 3. Banding is seen at 100X in all three materials; it is weakest in the 3-in. electrode stock (Figure 3a) and strongest in the powder-derived material (Figure 3c). The banding of the powder-derived material is strong enough to be seen at 1000X (Figure 3d), a magnification where banding is not seen in the other as-extruded materials.

Banding in the heat-treated tubes follows a similar pattern. In tubes aged directly at 950 F (Figure 4), banding is absent in the electrode stock but marked in the powder-derived tube. Resolutionizing and then aging (Figure 5) resulted in

some banding in the electrode stock but the banding is much more marked in powder material. In both materials, heavier or coarser precipitation is seen as a result of the resolutionizing (Figure 5 versus Figure 4).

The structures of the as-extruded rods shown in Figure 6 are similar to those of the tubes shown in Figure 3. The lower temperature extrusion of powder, if anything, results in less banding (Figure 6b). In the aged rods, banding is barely visible in the electrode stock at 100X (Figure 7a) and not seen at 1000X (Figure 7b), but is marked in the powder stock (Figures 7c and 7b). As in the tubes (Figure 3b), the heavier c/w stock (Tube B rear or B4 and Rod 5, not shown) shows banding intermediate to that of the electrode stock and the powder.

Table 3. MECHANICAL PROPERTIES OF STRIPS FROM 350 MARAGING STEEL TUBES

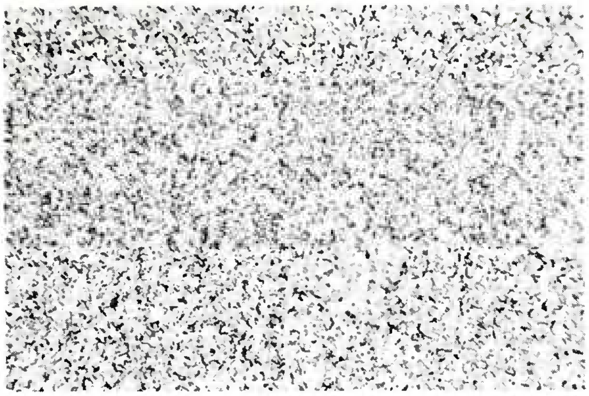
Tube Identification*	Material	0.2 Y.S. ksi	U.T.S. ksi	R.A. %	HRC	H Content ppm
B2A	Cast/wrought	345.7	347.3	45.2	58.2	
B2B	3-in.-diam	326.1	332.5	43.8	56.9	
B2C	bar (electrode	324.9	331.5	41.0	57.5	
B2D	stock)	348.6	352.2	36.5	58.1	
B4A	Cast/wrought	344.9	355.3	35.7	59.3	0.1
B4B	4-1/2-in. RCS	344.4	348.3	45.3	56.8	0.1
B4C	bar	350.6	352.4	39.0	58.0	
B4D		344.7	350.0	30.9	58.5	
E2A2	Rotating	353.5	353.6	25.2	58.8	0.2
E3A1	electrode	329.6	351.4	17.6	58.2	1.0
E3A2	powder	346.2	352.0	13.8	58.3	0.4
E2B		329.2	344.1	20.9	57.7	0.2
E2C		329.1	333.2	25.8	56.8	
E2D		343.2	349.6	25.6	57.7	

*First letter refers to tube, second letter is code for heat treatment
A Age 6 hours at 950 F
B Age 3 hours at 950 F
C Age 6 hours at 900 F
D Solutionize one hour at 1500 F; age 6 hours at 950 F

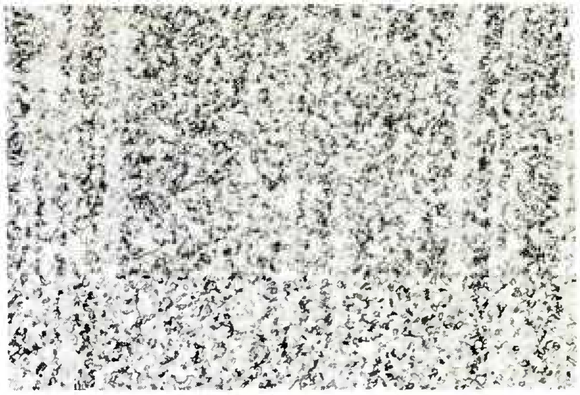
Table 4. MECHANICAL PROPERTIES OF 350 MARAGING STEEL RODS

Rod Identification*	Material Source	0.2 Y.S. ksi	U.T.S. ksi	Elon. %	R.A. %	Impact Resistance Charpy V, ft-lb R.T.	-40 C	HRC
2A	Cast/wrought	363.0	369.0	8.5	36.0	8.1	5.0	59.2
2D	3-in. dia. bar (electrode stock)	365.0	370.5	6.0	20.5	6.4	6.2	59.4
5A	Cast/wrought	348.5	359.0	7.5	32.5	8.1	5.4	58.3
5D	4-1/2 in. RCS bar	350.0	355.5	8.0	27.5	8.4	5.2	58.7
3A	REP powder	344.0	360.0	8.0	25.0	8.4	8.1	58.2
3D	ext at 1650 F	353.5	358.0	5.0	12.0	8.6	6.2	58.5
1A	REP powder	350.0	353.3	4.5	14.0	8.6	8.4	58.1
1B	ext at 1550 F	343.0	345.0	7.5	25.4	7.0	8.1	57.7
1C		336.0	345.0	8.5	19.5	8.1	5.2	57.7
1D		359.0	362.0	4.5	17.5	8.6	7.8	58.3

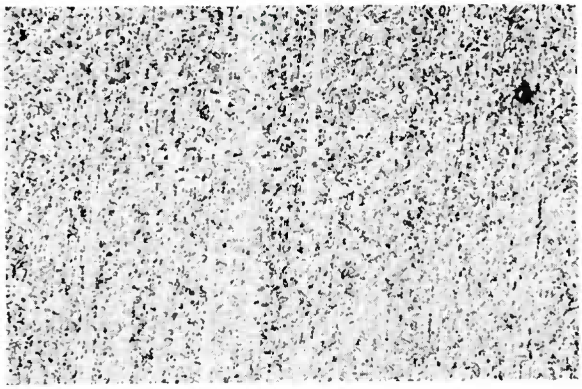
*Number refers to rod, letter is code for heat treatment
A Age 6 hours at 950 F
B Age 3 hours at 950 F
C Age 6 hours at 900 F
D Solutionize one hour at 1500 F; age 6 hours at 950 F



a. Electrode stock. Mag. 100X



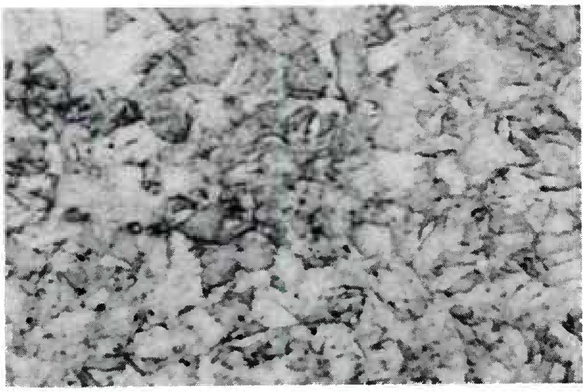
b. 4½-inch RCS bar stock. Mag. 100X



c. Powder stock. Mag. 100X

d. Powder stock. Mag. 1000X

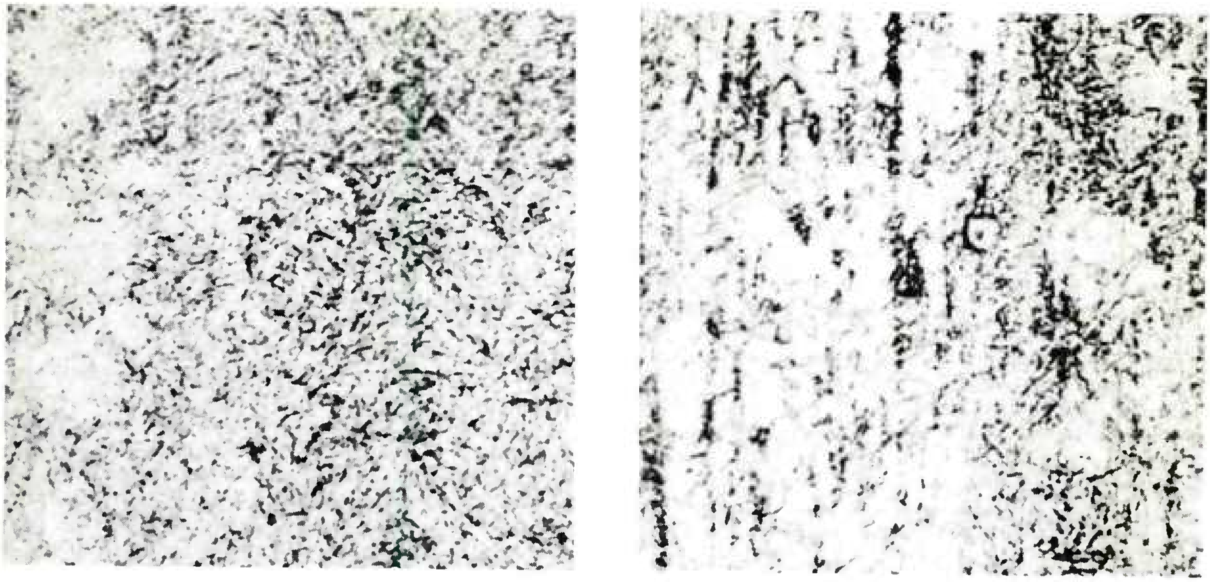
Figure 3. As-extruded 350 maraging tubes.



a. Electrode stock (B2A).

b. Powder stock (E3A1).

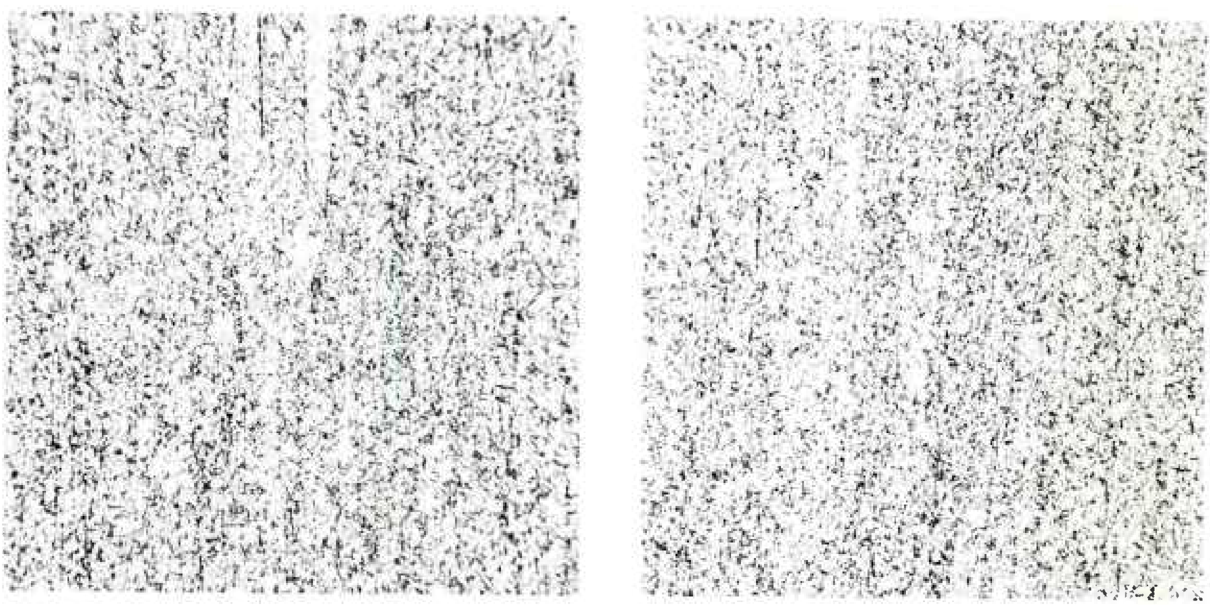
Figure 4. Heat-treated 350 maraging tubes, aged 6 hours at 950 F. Mag. 1000X



a. Electrode stock (B2D).

b. Powder stock (E2D).

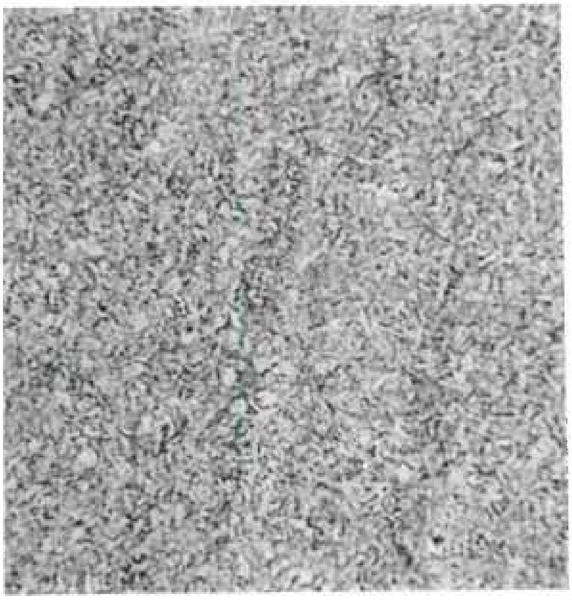
Figure 5. Heat-treated 350 maraging tubes resolutionized 1 hour at 1500 F, aged 6 hours ar 950 F. Mag. 1000X



a. Extruded at 1650 F (Rod 3).

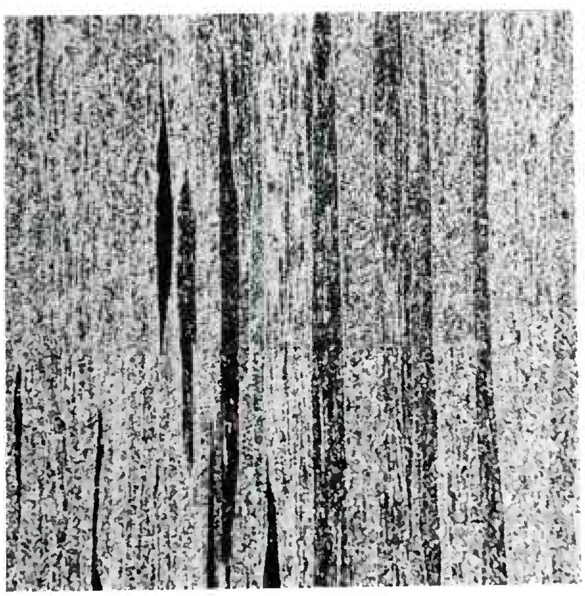
b. Extruded at 1550 F (Rod 1).

Figure 6. As-extruded 350 maraging rods, powder stock. Mag. 100X



a. Electrode stock (Rod 2). Mag. 100X

b. Electrode stock (Rod 2). Mag. 1000X



c. REP stock (Rod 3). Mag. 100X

d. REP stock (Rod 3). Mag. 1000X

Figure 7. Aged 350 maraging rods, aged 6 hours ar 950 F.

DISCUSSION OF RESULTS

The test data in Tables 3 and 4 permit assessment of the factors affecting the mechanical behavior of the tubes and rods. These assessments consider strength, ductility, and impact resistance. Hardness values are not cited, but in practically every case they support conclusions based on strength. Yield and tensile srengths are discussed interchangeably; their values are quite close, as the maraging steels undergo very little work hardening.

Heat Treatment

The specimens were subjected to the four heat treatments described earlier. Aging for 6 hours at 950 F (Heat Treatment A) is necessary to achieve full strength. This need was established for tubes from the electrode stock B and from powders E2 and E3, but not from the 4-1/2-in. bar stock B4. This need was confirmed for Rod 1, the only rod subjected to the full set of heat treatments. Reduction of time to 3 hours (Heat Treatment B) or temperature to 900 F (Heat Treatment C) left the material underaged, preventing realization of full strength (except for the B4 stock). This underaging did not affect impact resistance nor, except possibly for Rod 1, did it enhance ductility.

Resolutionizing (Heat Treatment D) before the optimum aging did not affect the strength of the tubes or rods made from either c/w or powder stock. The only exception is Rod 1 (lower temperature extrusion), whose strength was greater by 9 ksi after Heat Treatment D. The elongation and reduction of area data indicate that ductility may be lowered by the insertion of resolutionizing between extrusion and aging. The Charpy impact data are all so close that they do not show any effect of this resolutionizing.

It is significant that conventional c/w material can be directly aged after the 1650 F extrusion, dispensing with the usual 1500 F solutionizing. More detailed examination of the effects of fabrication variables on such material may be worth pursuing.

Material Source

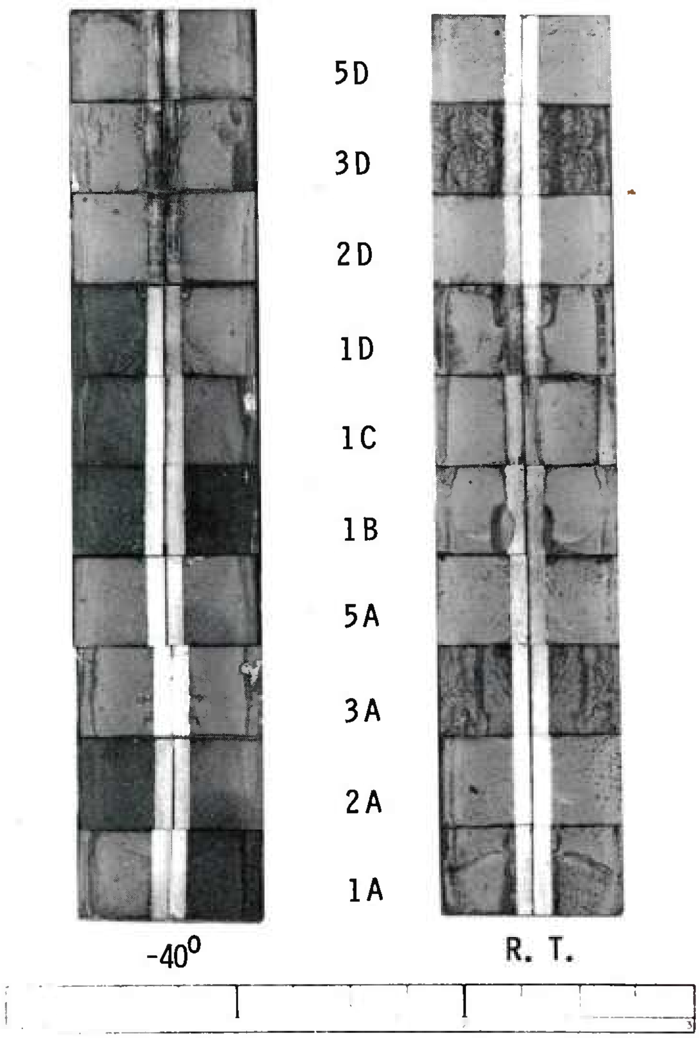
The comparison of c/w bars and powder as starting material was the principal point of interest in both the tubes and the rods. No evidence can be presented of greater strength as a result of the use of powder. In fact, the highest strength values (near 370 ksi) were observed in Rod 2, which was prepared from 3-in.-diameter electrode stock.

The powder-derived materials were lower in ductility (RA and also elongation, the latter available only for the rods). The low RA in some of the tubes may relate to another factor, hydrogen content, which was checked by analysis. The results in the last column of Table 3 are consistent with attribution of reduced ductility to hydrogen content,[11] perhaps with threshold content of about 0.4 ppm needed to degrade ductility. The hydrogen is not necessarily inherent to powder-derived material but may relate to the pickling of the tubes in concentrated acid. Attention is called to the lower hydrogen content and higher RA in tube stock E2 pickled in nitric acid versus stock E3 pickled in hydrochloric acid.

11. DAUTOVICH, D. P., and FLOREEN, S. *The Stress Intensities for Slow Crack Growth in Steels Containing Hydrogen.* Metallurgical Transactions, v. 4, no. 11, November 1973, p. 2627.

Greater significance is assigned to differences (or their absence) in impact resistance values for the various rods and also in the appearance of the fracture surfaces in both rods and tubes. The room-temperature Charpy values are indistinguishable - a remarkable numerical agreement in light of marked differences not only in material source but also in microstructure and appearance of the fracture surfaces. The subsequent Charpy data at -40 C differentiated somewhat between the materials. The powder-derived rods 1 and 3 hardly showed any difference from the room-temperature values; the c/w rods 2 and 5 decreased up to about 3 ft-lb, so that they were now lower than the powder-derived rods.

The impact specimens at both test temperatures showed smooth fracture surfaces in the c/w rods versus rougher, perhaps wooden surfaces in the powder rods (Figure 8). The difference in fracture surfaces is much more marked in the tension specimens (Figure 9). The c/w rods show a smooth cup/cone fracture in contrast to the wooden fracture of the powder rods. Differences resulting from material source and reflected in tensile fracture appearance might show up more strongly in other mechanical properties.



Number
1. REP - 1550 F
2. 3-inch bar
3. REP - 1650 F
4. 4½-inch RCS bar

Heat Treatment
A. 6 hours at 950 F
B. 3 hours at 950 F
C. 6 hours at 900 F
D. 1 hour at 1500 F; 6 hours at 950 F

Figure 8. Fracture surfaces of Charpy specimen (see Table 4 for rod specimen identification and properties).

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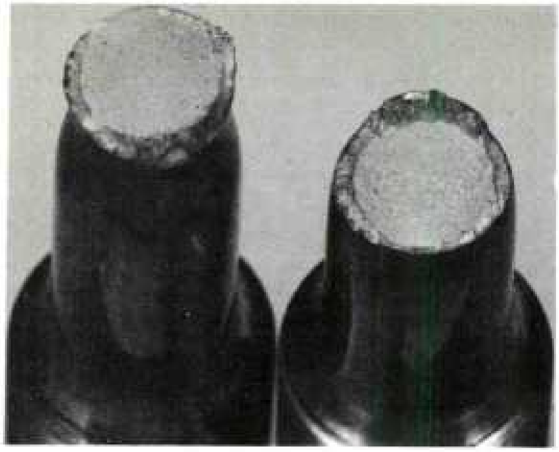

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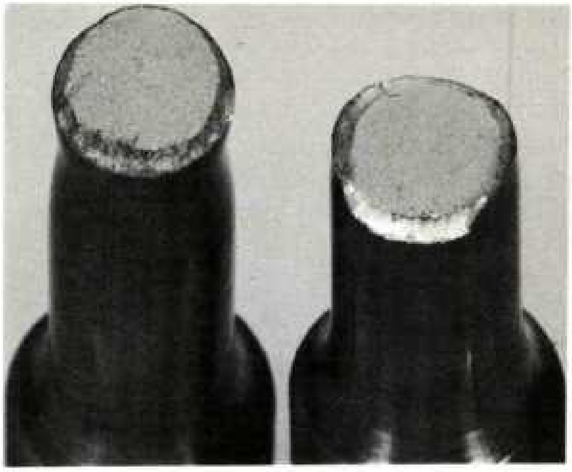
a. Aged c/w electrode stock 2A.



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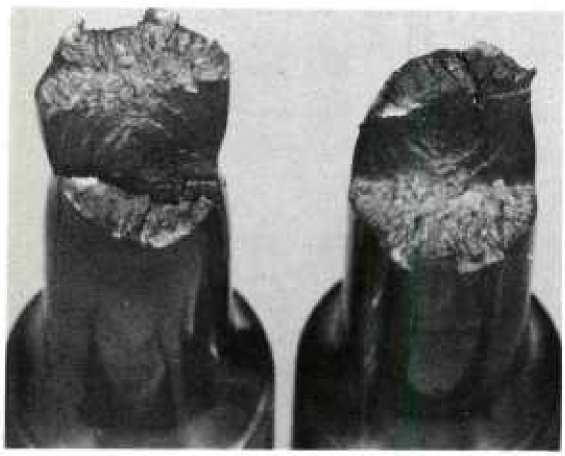
b. Solutionized and aged c/w electrode stock 2D.



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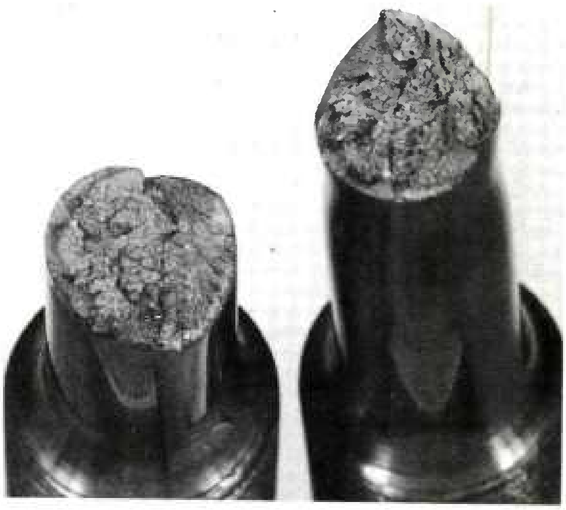
c. Aged powder stock 3A.



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d. Solutionized and aged powder stock 3D.

Figure 9. Fracture surfaces of tension specimens.

The outstanding strength of Rod 2 deserves separate discussion. The tubing B2 prepared from the same electrode stock did not stand out. Microstructurally the rod and the tube were similar (Figures 4a and 7b); they differed from all the other extrusions in the absence of banding. It remains to be resolved whether the rod or the tube is more representative of this stock. Conceivably the heavier extrusion reduction of the rod (13.1 versus 9.6) may have effected greater grain refinement. A possibility exists that the rods were extruded at lower true billet temperature, as suggested by the extrusion constants (Table 2). Differences between rods and tubes from the same stock are discussed in the next section. In no case were they anywhere near as large as between Rod 2 and Tube B2. Whatever the explanation, the outstanding strength of Rod 2 confirms that powder is not necessary for the very fine, strong 350 maraging steel. Such material can apparently be obtained by heavy working, which the 3-in. bar stock must have received. The benefits of heavy working of commercial 300 maraging stock have been cited by Van Swam et al.[10] More generally, these maraging steels already have such a fine distribution of microstructural features that little room remains for further contribution from powder processing.

Rods Versus Tubes

The data of Tables 3 and 4 permit direct comparisons between tubes and rods made from the same starting material: Tube B2 versus Rod 2; Tube B4 versus Rod 5; Tubes E2 and E3 versus Rod 3. The difference of about 20 ksi within the first pair (that is, the outstanding strength of Rod 2) has been cited above. For the other pairings, the rod is almost always stronger but only by about 5 ksi. This difference may be due to the higher reduction ratio (13:1) of the rods, possibly accompanied by a lower extrusion temperature as suggested by the unexpectedly higher extrusion constants. The achievement of yield strength of 345 ksi and ultimate strength of 350 ksi in all the tubes (when adequately aged) supports the expectation that extrusion can be scaled up for larger components.

Extrusion of Powder at Different Temperatures

Rods 1 and 3 provide the only comparison of the effect of different extrusion temperatures on the same starting materials. The overlap of the strength data prevents the drawing of any conclusion. Inferences regarding effect of the lower temperature of Rod 1 would have to be qualified by its more complicated schedule of being heated twice, the first time for an unsuccessful attempt to extrude at 1400 F.

SUMMARY AND CONCLUSIONS

Rods and tubes of 350 maraging steel were extruded from rotating electrode process powder concurrently with cast-and-wrought bar stock from two sources.

The various extrusions of rods and tubes from powder or bar stock were directly aged to full strength without an intermediate resolutionizing between extrusion and aging.

Aging for 6 hours at 950 F was necessary for achievement of full strength. Reduction of the time or lowering of the temperature gave underaged material, lower in strength and no better in ductility or impact resistance.

Strength levels were approximately the same for all the rods and tubes with the exception of one stronger rod prepared from bar stock. Ductility values (elongation and reduction of area) tended to be slightly lower for the powder-derived rods. Greater lowering of RA was found in the powder-derived tubes but this lowering may be due to another factor such as hydrogen pick-up during acid pickling.

Charpy values, determined only on rods, were hard to distinguish at room temperature. The values at -40 C showed a slight but distinct advantage for the powder-derived rods. This advantage is related to the different fracture modes, seen in both impact and tension specimens (Figures 8 and 9). These differences are related to the greater banding of powder-derived material. Such material might show up to greater advantage in other structure-sensitive properties, such as compressive strength, fracture toughness, fatigue, or stress corrosion resistance.

The rod prepared from one type of bar stock (used for electrodes for the REP powder) manifested exceptional strengths (near 370 ksi) in the tension tests. Such strength is presumably the result of heavier working of the starting material, also indicated by the absence of banding in the extrusions (both rod and tube). The tube prepared from this stock resembled tubes prepared from the other materials in showing strengths near 350 ksi.

The powder approach may be more effective in enhancing the properties of other grades of maraging steel such as the nominal 400 grade (see Appendix). Since this grade really has strengths closer to the 380 ksi level, the value of the 400 approach (heavier alloying with Co and Mo) has to be weighed against the benefits obtainable from better understanding of the 370 ksi rods prepared here from heavily worked bar stock.

Tubes with strengths very close to expected levels have been successfully prepared. The results indicate that the process can be scaled up for larger tubes. Such scale-up may be applicable to cast-and-wrought stock at a lower reduction ratio than needed for powder consolidation. The required press capability for larger tube extrusion could thus be kept within bounds permitted by more available presses. Larger tubes would be suited for more extensive characterization without introducing the need for the tube flattening used here.

ACKNOWLEDGMENT

Appreciation is expressed to Mr. Charles Nolan of Watervliet Arsenal for providing the 4-1/2-in. RCS bar stock and making his experience with the maraging steels available in some very helpful discussions.

APPENDIX. 400 MARAGING STEEL

It is reasonable to extend the maraging approach to obtain even stronger steels than the 350 grade while retaining adequate toughness for structural application. Earlier grades have developed in increments of 50 from 250 to 300 to 350 where the designation represents tensile or yield strength in thousand pounds per square inch (these strengths are not far apart, as the maraging steels undergo very little work hardening). It is thus natural to designate the next grade as 400, although, as will be seen below, the 400 ksi level has been reached only in limited cases, and a number like 380 is more realistic.

Early workers did report strengths near 400 ksi with modest ductilities. In more recent definitive work, Magnée et al.[12] reached a limit of about 380 ksi. Their best combination of strength with toughness was at a strength level of 372.6 ksi ultimate and 367.0 ksi yield. At this strength, the fracture toughness was higher than that of 350 maraging. In spite of these improvements over 350, the "400" grade has not gained acceptance as a commercial material. Presumably the gains over 350 are not sufficient to justify departure from the well-established and available 350 grade.

Abrahamson's work on REP 400 may now be judged in better context.[7] He brought the strengths to the 380 level with an R.A. of 15% versus the 6.4% observed by Magnée in the high-toughness material (K_{Ic} 55 ksi $\sqrt{\text{in.}}$). Conceivably a 380 grade achieved through the powder approach may be worth pursuing for some applications.

12. MAGNEE, A., VIATOUR, P., DRAPIER, J. M., COUTSOURADIS, D., and HABRAKEN, L. *Microstructure, Strength and Toughness of 13Ni (400) Maraging Steel.* Cobalt, 1973-1, p. 3.

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Toughness

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